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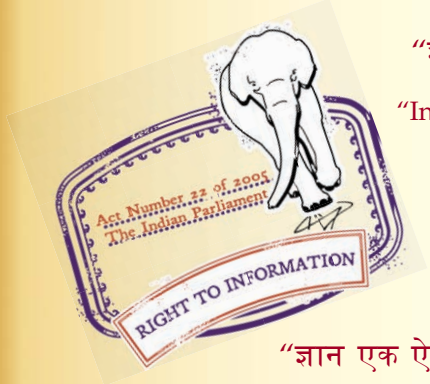
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IS 3660-9 (1989): Methods of Test for Natural Rubber, Part 9: Determination of Solvent Extract [PCD 13: Rubber and Rubber Products]

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IS 3660 (Part 9) : 1989

(Reaffirmed 2000)

Indian Standard

METHODS OF TEST FOR NATURAL RUBBER

PART 9 DETERMINATION OF SOLVENT EXTRACT

NR : 10

(First Revision)

भारतीय मानक

प्राकृतिक रबड़ की परीक्षण विधियाँ :

भाग 9 विलायक निष्कर्ष का निर्धारण

एन आर : 10

(पहला पुनरीक्षण)

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Price Group 2

FOREWORD

This Indian Standard (Part 9) (First Revision) was adopted by the Bureau of Indian Standards on 14 April 1989, after the draft finalized by the Rubber Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Methods of test for natural rubber had been originally covered in the following four parts of IS 3660:

Part 1 : 1972 Determination of dirt, volatile matter, ash, total copper, manganese, rubber hydrocarbon, viscosity (shearing disc viscometer), and mixing and vulcanizing of rubber in a standard compound

Part 2 : 1968 Determination of solvent extract and nitrogen content

Part 3 : 1971 Plasticity and plasticity retention index

Part 4 : 1979 Determination of colour, accelerated storage-hardening test and vulcanization characteristics (MOD test)

While reviewing various test methods for natural rubber, the Committee decided to align them with the corresponding international standards. No unification of test methods for natural and synthetic rubber has been considered necessary. However, in revising test methods for natural rubber, the Committee had decided to revise and split the standard into further parts and publish individual test methods under natural rubber (NR) series. For proper referencing of the existing test methods and the new methods under revision, a table showing correspondence of the various methods of test covered in the previous 4 parts of IS : 3660 (Parts 1, 2, 3 and 4) with the presently split parts retaining the original NR : number have been given in the table given in Annex A.

In order to facilitate cross-reference, it has been decided to retain the original discrete NR series numbers assigned to various test methods in original IS : 3660 (Parts 1, 2, 3, and 4) in the revised parts of IS : 3660.

The test method given in this revised standard will supersede the test method given under NR : 10 of IS : 3660 (Part 2) : 1968. All the parts of the original IS : 3660 shall be withdrawn upon its complete revision.

In the preparation of this standard, assistance has been derived from ISO/DIS 1407 'Rubber — Determination of solvent extract', issued by the International Organization for Standardization (ISO) which prescribes two methods for quantitative determination of extractable material from raw rubber (both natural and synthetic), namely, Method A using a Soxhlet — type apparatus and Method B rapid evaporation method by boiling the sample in appropriate solvent without using Soxhlet. However, in the revision of this Standard, only one method which is similar to method A of ISO/DIS has been prescribed. Method B of ISO/DIS has not been included since it generally gives lower results than Method A due to an equilibrium being set up, particularly if a large sample mass is used.

In this revision, based on ISO/DIS 1407, the following modifications have been made:

a) Number of extraction has been changed from 'minimum 40 extraction for 8 to 16 h' to '10 to 20 extraction per hour for 16·0 ± 0·5 h'.

b) Calculation has been modified by including 'blank correction factor'.

c) An additional type of apparatus (Fig. 3) has been included.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS OF TEST FOR NATURAL RUBBER

PART 9 DETERMINATION OF SOLVENT EXTRACT

NR : 10

(First Revision)

1 SCOPE

1.1 This standard (Part 9) prescribes method for determination of solvent extract of raw natural rubber.

2 REFERENCES

2.1 The Indian Standards listed below are necessary adjuncts to this standard:

IS No.	*Title*
IS 170 : 1986	Specification for acetone (*third revision*)
IS : 3660 (Part 1) : 1972	Methods of test for natural rubber : Part 1 Determination of ash, total copper, manganese, rubber hydrocarbon, viscosity (shearing disc viscometer), and mixing and vulcanizing of rubber in a standard compound (*first revision*) [*under revision* as IS 3660 (Part 16) (NR : 17)]

3 OUTLINE OF THE METHOD

3.1 Extraction of an individual weighed test portion of the rubber is carried out in Soxhlet-type apparatus with the appropriate solvent (acetone). The solvent is then removed from the extract by distillation or evaporation, followed by drying and weighing of the residue.

4 APPARATUS

4.1 Extraction Apparatus

Three types of extraction apparatus are suitable. Any other type of apparatus which performs the same function may be used.

a) *Type 1*, comprising of a receiver flask, a jacketed Soxhlet extractor and a condenser as shown in Fig. 1.

b) *Type 2*, comprising of a 250-ml receiver flask, a condenser and an extraction cup suspended by clean wire as shown in Fig. 2.

c) *Type 3*, comprising of a 250-ml receiver flask, a condenser and a Soxhlet extractor with a side arm as shown in Fig. 3. Typical Soxhlet capacity is 100 to 200 ml. There is no extraction cup.

4.2 Distillation Head and Condenser

It shall be appropriate for the apparatus. It is used to distil off the solvent after extraction.

4.3 Oven, operating at 75 ± 2°C or 100 ± 2°C, as required.

4.4 Filter Paper or Nylon Filter Cloth, extracted before use, with the same solvent used for the extraction.

5 REAGENT

5.1 Acetone, conforming to IS 170 : 1986.

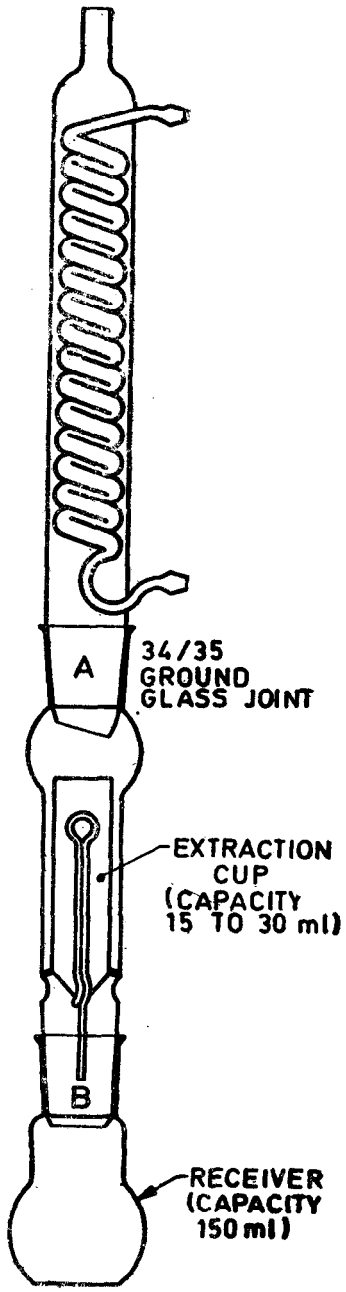
6 PROCEDURE

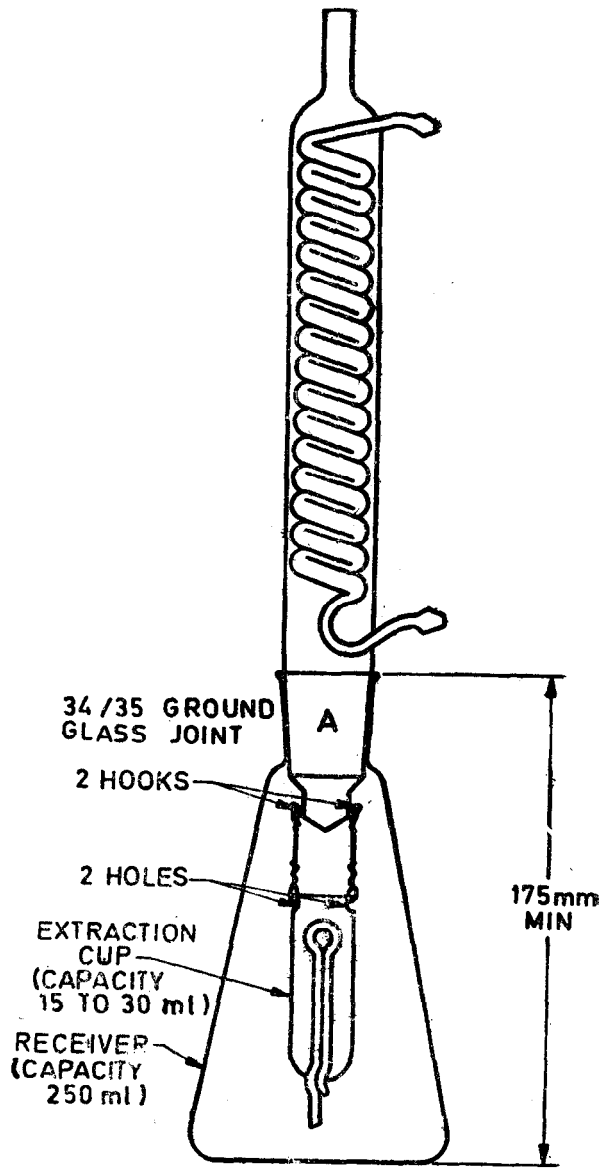
6.1 Cut a portion of the rubber weighing about 5 to 10 g from the homogenized piece as prepared in 3 of IS 3660 (Part 1) : 1972. Pass the rubber through the cold rolls of a laboratory mill set to a nip not exceeding 0·5 mm.

It is convenient to run the milled sheet into polyethylene or glazed linen to prevent sticking as it leaves the mill. Cut from the sheet, avoiding the edges, a test portion estimated to weigh 3 g and weigh to the nearest 0·1 mg.

6.2 Dry the chosen flask in an oven at 100 ± 2°C. Remove the flask from the oven and allow it to cool to room temperature in a desiccator. Weigh to the nearest 0·1 mg.

6.3 Roll the milled, weighed test portion in filter paper or nylon cloth to form a loose roll from which the rubber does not fall and so that no part of the rubber is anywhere in contact with any other part of the rubber. If the test portion is in the form of small pieces, make a loose packet of the pieces in filter paper or nylon cloth. Fasten either test packet with clean wire. Place the packet in the appropriate extraction apparatus, and pour in sufficient acetone to fill the extraction cup.





Extraction Cup Capacity	Joint		Receiver Capacity
ml	*A*	*B*	ml
20 to 30	34/35	34/35	150
50 to 60	45/40	34/35	250

FIG. 1 TYPE 1 ALL GLASS EXTRACTION APPARATUS

FIG. 2 TYPE 2 ALL GLASS EXTRACTION APPARATUS

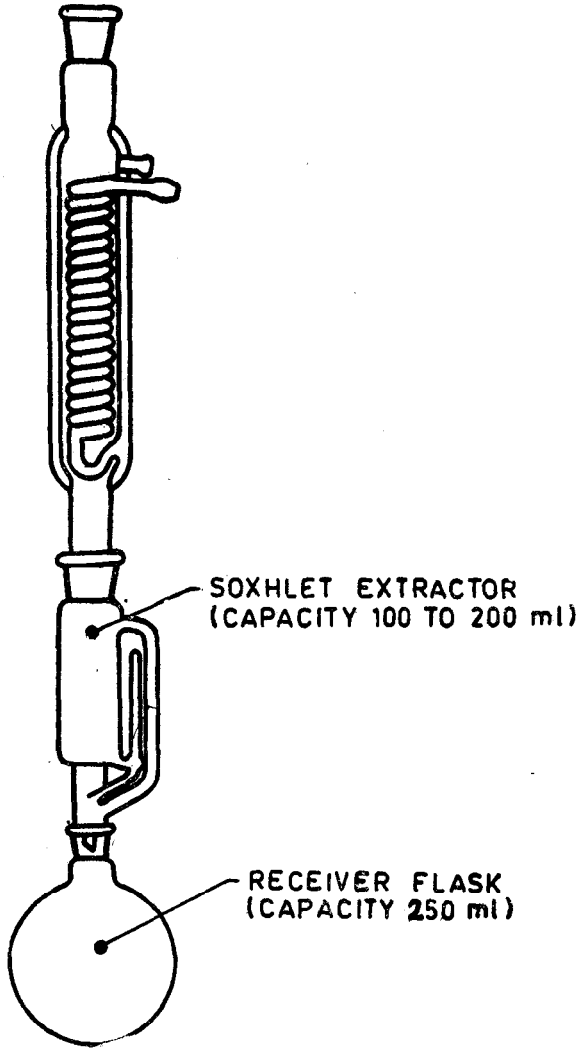


FIG. 3 TYPE 3, ALL GLASS EXTRACTION APPARATUS

Assemble the apparatus and adjust the heating rate so that the quantity of distilled solvent fills the extraction cup 10 to 20 times per hour and extract for 16·0 ± 0·5 h.

6.4 At the end of the heating period, turn off the heating device, allow the apparatus to cool, then remove the extractor or siphon cup and discard the rubber test portion unless it is required for further testing.

6.5 Remove the receiver flask, fit it with the distillation head and condenser and distil off the bulk of the solvent into a suitable flask, retaining about 5 ml of solvent in the flask. Alternatively, a rotary evaporator may be used. Discard the distillate unless it is required for further testing.

NOTE — The solvent may also be evaporated from the open flask by gentle heating with the heating device. This may be done only where local health and safety regulations permit and only in a well-ventilated fume cupboard.

6.6 Dry the flask and contents for 2 h at 100 ± 2°C in the oven and at the end of this time, remove the flask from the oven, cool in a desiccator and weigh to the nearest 0·1 mg.

6.7 Carry a blank through the entire procedure, using the same type of apparatus and quantity of solvent as for the test portion.

7 CALCULATION

7.1 The solvent extractable material, expressed as percent by mass, is given by the formula:

Solvent extract,

$$\text{percent by mass} = \frac{m_2 - m_1 - m_3}{m_0} \times 100$$

where

m_1 = mass in g of the empty receiver flask,

m_2 = mass in g of the receiver flask plus the extract after drying,

m_3 = increase in mass in g of a receiver flask during the blank test, and

m_0 = mass in g of the test portion.

8 TEST REPORT

8.1 The test report shall include the following particulars:

a) Full identification of the rubber tested,

b) Reference to this standard,

c) Method of sample preparation,

d) Solvent used,

e) Which apparatus was used for extraction, and

f) Mean of two determinations.

ANNEX A

(Ref : *Foreword*)

Table showing Correspondence of the various methods of test covered in the existing IS 3660 (Part 1) : 1972, IS 3660 (Part 2) : 1968, IS 3660 (Part 3) : 1971, and IS 3660 (Part 4) : 1979 with the revision/ proposed revision of all the four Parts of IS : 3660

Existing Test Methods			*Proposed Revision*		*Remarks*
Test Method	IS No.	Part (Series)	IS No.	Series	
NR SERIES					
Determination of dirt	IS 3660 : 1972	Part 1 (NR : 1)	IS 3660 (Part 1) : 1985	(NR : 1)	
Determination of volatile matter	IS 3660 : 1972	Part 1 (NR : 2)	IS 3660 (Part 2) : 1985	(NR : 2)	
Determination of ash	IS 3660 : 1972	Part 1 (NR : 3)	IS 3660 (Part 3) : 1988	(NR : 3)	
Determination of total copper	IS 3660 : 1972	Part 1 (NR : 4)	IS 3660 (Part 4) : 1988	(NR : 4)	
Determination of manganese	IS 3660 : 1972	Part 1 (NR : 5)	IS 3660 (Part 5) : 1989	(NR : 5)	
Determination of iron	IS 3660 : 1972	Part 1 (NR : 6)	Deleted since this test is no longer being done		
Determination of rubber hydrocarbon	IS 3660 : 1972	Part 1 (NR : 7)	IS 3660 (Part 6) : 1988	(NR : 7)	
Determination of viscosity by shearing disc viscometer	IS 3660 : 1972	Part 1 (NR : 8)	IS 3660 (Part 7) : 1988	(NR : 8)	
Mixing and vulcanizing in a standard compound	IS 3660 : 1972	Part 1 (NR : 9)	IS 3660 (Part 8)	(NR : 9)	Under revision
Determination of solvent extract	IS 3660 : 1968	Part 2 (NR : 10)	IS 3660 (Part 9) : 1989	(NR : 10)	
Determination of nitrogen content	IS 3660 : 1968	Part 2 (NR : 11)	IS 3660 (Part 10)	(NR : 11)	Under revision
Determination of plasticity	IS 3660 : 1971	Part 3 (NR : 12)	IS 3660 (Part 11) : 1989	(NR : 12)	
Determination of plasticity retention index (PRI)	IS 3660 : 1971	Part 3 (NR : 13)	IS 3660 (Part 12) : 1989	(NR : 13)	
Determination of colour	IS 3660 : 1979	Part 4 (NR : 14)	IS 3660 (Part 13)	(NR : 14)	Under revision
Determination storage-hardening test	IS 3660 : 1979	Part 4 (NR : 15)	IS 3660 (Part 14)	(NR : 15)	
Determination of vulcanization characteristics (M O D test)	IS 3660 : 1979	Part 4 (NR : 16)	IS 3660 (Part 15)	(NR : 16)	
Method for preparation of test samples	IS 3660 : 1972	Part 1 (clause 3)	IS 3660 (Part 16)	(NR : 17)	

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